Laboratory Continuous Deodorizer for Vegetable Oils[1]

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A laboratory-scale continuous deodorizer, based on a modified Snyder distillation column, was constructed and tested for the deodorization of alkali-refined and bleached vegetable oils. Soybean oil extracted with supercritical carbon dioxide and without further processing also was deodorized to a finished edible oil. Results of taste panel evaluations of the finished oils show that the quality of oils deodorized over a temperature range of 194-260 C is equivalent to commercial salad oils. Oil flow rates are 1 to 2 ml/min, and contact time is about 5 min; a vacuum of 0.5 to 1.0 mm Hg is maintained with countercurrent steam flow of 1 to 5% of the oil weight. Small samples of oil (250-1000 ml) are readily accommodated in this equipment, and the deodorization conditions more nearly simulate commercial practice than do traditional small-scale batch deodorizers.

Continuous or semicontinuous deodorization of vegetable oils is the accepted practice in commercial applications (1,2). These deodorizers are designed to accomplish deaerating, heating, stripping and cooling (2-4). Some of the latest commercial continuous operations vary the oil depth from a thin layer (5-7) to an atomized spray (8), and temperatures range from about 200 C to 260 C.

In the laboratory, batch type deodorizers are still commonly used because only small amounts of oil can be accommodated (9). No laboratory deodorizers are of the continuous type, although some are semicontinuous in operation (10). However, none of the laboratory deodorizers adequately simulates present industrial practice or lends itself readily to other uses, such as evaluation of deodorization parameters, variation of sample size and testing of transducers and sensors for computer control applications.

APPARATUS

A diagram of the apparatus is shown in Figure 1. The system consists of a modified Snyder glass distillation column (Ace Glass Inc., Vineland, New Jersey) containing 19 bubble-cap stages (see inset BB for details of stages). Two sidearms were added, one for steam inlet and the other to permit the use of an internal thermocouple. A glass adapter was fitted to the top of the column to accommodate a vacuum attachment, oil inlet and oil pre-heat area containing glass beads (see inset AA for details). (The oil reservoir is equipped with a teflon-glass needle valve for control of the oil flow rates.) Configuration of the oil reservoir allows oil to be added and deaerated during a run. Adapters at the base of the column permit taking finished oil or oil samples without interrupting operation of the column. Sample size is limited by oil flow rate to about 500 ml for a working day. A graduated separatory funnel was modified, by removing the bottom stopcock and placing a high

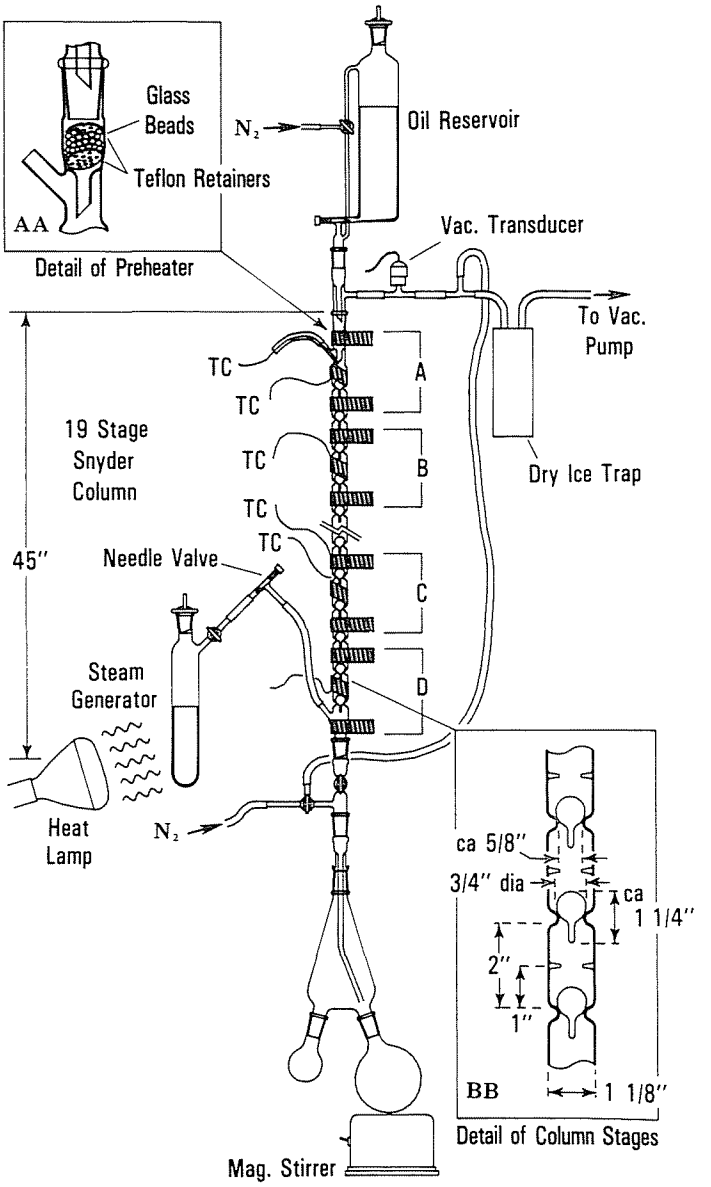

FIG. 1. Continuous deodorizer. Inset AA—enlarged view of glass bead pre-heat area. Inset BB—enlarged view of Snyder column stages. A, B, C, D—column heaters. TC—thermocouples. N_2—nitrogen gas inlet. VAC—vacuum. See text for further details.

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vacuum stopcock in the outlet arm, to form the steam generator. A teflon-glass needle valve is positioned in the steam line for more precise setting of steam flow. No external heat is required for steam generation when the room temperature is ca. 25 C. An infrared lamp with controller is used for heating the water when needed. Bumping or "burping" of water in the steam generator did not occur. Four heaters (A-D) are used to heat the column, and six thermocouples measure inside and outside temperatures. The bottom (D) and top (A) heaters serve dual roles: in addition to heating the column, the bottom heater keeps the steam from condensing as it enters the column, and the top heater preheats the oil by controlling the temperature of the glass beads (inset AA-Fig. 1). It is absolutely essential that no standard taper glass joints be subjected to deodorization temperatures because of the danger of air leakage into the system.

Accessory units are not shown in Figure 1; they consist of a five-position digital meter for external temperature measurement, a potentiometer or millivoltmeter for measurement of the internal temperature, four variacs for heater control, a thermistor-type vacuum meter with sensors (General Electric, Model No. 22GC320), a magnetic stirrer for incorporating antioxidant, and a standard vacuum pump.

PROCEDURE

All of the electronic equipment is turned on and allowed to reach equilibrium. About 30 min is required for the column to reach deodorization temperature (260 C). During this 30 min, the steam generator is charged with distilled water and oil is added to the reservoir and deaerated. After initiating steam flow and attaining a temperature of 260 C, the oil reservoir needle valve is adjusted to give an oil flow rate of about 1 ml/min. This valve needs to be adjusted slightly as the deodorization proceeds to maintain constant oil flow rate because of the change in head pressure in the oil reservoir. The time elapsed between the start of oil flow and the time oil appears at the bottom of the column is the residence time. Residence time for soybean oil is 4 to 5 min. No adjustments other than to maintain oil flow rate are required during deodorization. However, if oil flow rate is changed, adjustment of the top heater is necessary to maintain operating temperature. Although the deodorizer requires little attendance during operation, all measurements have to be recorded every 30 min and steam and oil flow rates must be calculated to monitor the run accurately. If desired, antioxidant can be added to the deodorized oil and the mixture stirred under vacuum for 30 min. After the entire system has returned to room temperature, the column is cleaned by flushing with hexane and air dried, and the deodorizer distillate is removed from the dry ice trap.

Normally, analyses for phosphorus, iron, peroxide value (PV) and free fatty acid (FFA) are performed on all oils before and after deodorization (11). Color of the deodorized oils is determined (11). Finished oils are evaluated for initial flavor and flavor stability (12).

RESULTS AND DISCUSSION

Soybean oil, corn oil, cottonseed oil and jojoba wax esters have been processed successfully in the continuous deodorizer. Only the results achieved with soybean oil are given here for illustration. Varying temperatures between 200 C and 260 C (Table 1) had no significant effect on flavor of the oil but dramatically affected its color. These color differences are shown in Table 1. As expected, the highest temperature (260 C) produced the lightest oil (13). According to the literature (2), an acceptable deodorized soybean oil would have a color of 10Y 0.7R or lighter. Our apparatus produces an acceptable oil color at temperatures between 230 C and 260 C with 1% to 3% steam flow at 0.4 to 1.0 torr vacuum.

Vegetable oils extracted by hexane and by supercritical carbon dioxide (SC-CO_2) were processed to finished deodorized oils, analyzed, and evaluated by a flavor panel. SC-CO_2 extracted oil was divided into two portions; one portion was mixed with Celite® and filtered, but there was no pretreatment of the second portion. The oils were again divided into two portions, so that a total of four deodorizations was performed. All oils were treated with Tenox 20 (Eastman Chemical Co.). Results of chemical analyses of finished oils processed from SC-CO_2 extracted crude oils were not significantly different. There were no significant differences in flavor evaluation due to the extraction process for soybean, corn and cottonseed oils. Table 3 shows the results of flavor evaluation of SC-CO_2 extracted, laboratory deodorized soybean oil and commercial refined-bleached-deodorized soybean oil. There was no significant difference between flavor scores of the commercial oil and the four oils extracted

TABLE 1

Effect of Deodorization Temperature on Color of Soybean Oil[a]

Temperature (°C)	Lovibond Color	
Starting Oil[b]	50Y	3.5R
200	35Y	0.7R
230	10Y	0.3R
240	6Y	0.2R
260	4Y	0.2R

[a]Deodorization conditions, 0.4-1.0 torr, 1-3% steam flow, 1.0 ml/min oil flow rate.

[b]Refined-bleached commercial oil.

TABLE 2

Analyses of Continuously Deodorized Soybean Oil Extracted with Supercritical CO_2[a]

	Celite-Filtered		No Treatment	
P.V. (meq/kg)	0	0	0	0
FFA (%)	0.06	0.05	0.05	0.06
Phos (ppm)	0.3	0.2	0.2	0.1
Fe (ppm)	0.62	0.60	0.57	0.70
Color	9Y 0.1R	10Y 0.1R	10Y 0.1R	10Y 0.5R

[a]Deodorization conditions, 260 C, 0.4-1.0 torr, 1-3% steam flow, 1.0 ml/min oil flow rate.

TABLE 3

Flavor Evaluation of Continuously Deodorized Soybean Oil Extracted with Supercritical CO_2[a,b]

Storage at 60 C (days)	Deodorized Commercial Oil	CO_2 Extracted Crude			
		Celite-Filtered		No Treatment	
0	7.1	7.4	8.0	6.8	7.1
4	6.0	5.6	6.7	6.1	6.0

[a]Rating scale of 10: 1, strong; 10, bland.

[b]Deodorization conditions, see Table 2.

with SC-CO_2. Thus, steam deodorization of SC-CO_2 extracted crude soybean oil, without any prior refining, produces oil equivalent to commercially deodorized oils.

Laboratory continuous deodorizers of the type described here have several useful applications. They can be incorporated into a minirefinery (14) which more nearly simulates industrial practice, they serve as a means of testing transducers and sensors for computer control applications (15,16), and they can be used to produce research-sized samples for organoleptic evaluation of oils.

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